

CH 369,107

Job No.: 1505-88657

Translated from French by the Ralph McElroy Translation Company
910 West Avenue, Austin, Texas 78701 USA

SWISS CONFEDERATION
FEDERAL OFFICE FOR INTELLECTUAL PROPERTY
INVENTION DISCLOSURE
PATENT NO. 369107

Classification:	8m, 1/01
Filing No.:	74898/59
Filing Date:	October 1, 1959, 12 midnight (Article 115, paragraph 2 LBI)
Priority:	USA, June 26, 1958 (744676) (effect limited by article 115, paragraph 2 LBI)

PROCESS FOR IMPROVING THE FASTNESS OF DYES OR PRINTS ON TEXTILE
MATERIALS

Inventor:	Domenick Donald Gagliardi, East Greenwich, Rhode Island, USA
Application Published:	January 31, 1963
Patent Granted:	May 15, 1963
Invention Disclosure Published On:	June 29, 1963
Corresponding to Disclosed Description No.	74898/59
[Applicant]:	Principal patent Union Carbide Corporation, New York (USA)

The invention is based on the discovery that aminoalkyl-silicon compounds and the coordinated metallic complexes of these compounds, applied on natural or synthetic textile articles dyed or printed beforehand, improve the fastness of the dyes or prints, particularly the fastness with regard to washing.

It is preferable to apply the aminoalkyl-silicon compounds by immersion of the dyed or printed article in an aqueous solution of these compounds, but it is completely impossible [sic] to

use spraying or padding. It is also possible to use emulsions of the dye fixing agents satisfactorily. It is possible to promote a more uniform distribution of the dye fixing agent on the dyed or printed textile articles by using solvents which do not react with the aminoalkyl-silicon compound or by using wetting agents.

Among the solvents which can be used to promote more uniform distribution of the dye fixing agent, it is possible to mention alkanols and ether-alcohols such as ethanol, propanol, isopropanol, methoxyethanol, and ethoxyethanol. As solubilizing agents which can be used successfully, it is possible to mention formic, acetic, propionic, lactic, gluconic, glycolic, and diglycolic acids.

It is also possible to use mineral acids as solubilizing agents, and as solvent, it is possible to use solvent aromatic hydrocarbons such as benzene, toluene and xylene. Finally, it is possible to deposit the dye fixing agents using aqueous alkaline solutions.

An aqueous system containing 40 to 60 parts water, 40 to 60 parts organic alcohol such as ethanol or isopropanol and 5% by volume of a monobasic acid such as acetic acid is an excellent medium for solubilizing and dispersing the aminoalkyl-silicon compounds on practically any dyed or printed textile material.

The concentration of the aminoalkyl-silicon compounds in the treatment solutions can be between 1 and 5 wt%, and good fixing of the dye is obtained in the case of the majority of direct dyes, with quantities of deposited solids between 0.1 and 2.0 wt%.

The aminoalkyl-silicon deposits obtained are advantageously dried by heating the textile article. However, simple drying in air at room temperature can fix the dye in numerous cases in such a way as to provide good fastness with regard to washing.

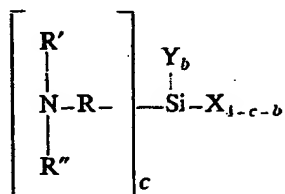
It is preferable to operate at heating temperatures between 95 and 180°C for periods of time between a few minutes and half an hour, depending on the particular colored textile article. When the fixing of the dye is done on a moist textile (dyed or printed), it is possible to incorporate the necessary drying in the heating cycles required for normal dyeing or printing, or it is possible to separate the thermal treatment from these normal operations.

As textile articles on which dyes can be fixed by the process according to the invention, it is possible to mention those made of cotton, flax, ramie, hemp, jute, cellulose ethers, cellulose esters, regenerated cellulose rayons, natural silk, tussore and wool.

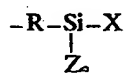
In addition to the treatment of conventionally dyed fibers, it is also possible to apply the process according to the present invention in order to improve the fixing of dyes which have been deposited on textile articles in the manner described in French Patent No. 1232590. This patent relates to the treatment of textile articles by aminoalkyl-silicon compounds before dyeing or simultaneously with it using pigments both of natural and synthetic origin or using anionic coloring agents, in order to improve the deposition of the coloring agent on the latter. The

fastness of these dyes and prints is improved by a later treatment of the textile article by an aminoalkyl-silicon compound according to the present invention.

The aminoalkyl-silicon compounds used, as well as their coordinated metallic complexes, ordinarily contain at least one group with formula:



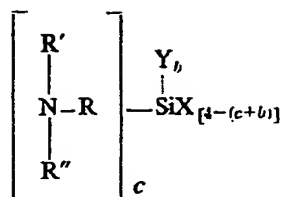
in which R is a substituted or non-substituted hydrocarbon group with at least 3 carbon atoms between the nitrogen atom and the silicon atom; R' and R'' represent hydrogen, alkyl, aminoalkyl, cyanoalkyl, hydroxyalkyl, carboalkoxyalkyl, aryl radicals or the monovalent group:



X is an alkoxy radical or the oxygen atom of a siloxylidyne radical $[\equiv \text{Si}-\text{O}-]$; Y is a hydroxy, alkyl or aryl radical; Z is an alkoxy, alkyl or aryl radical; c is 1 or 2; b is equal to 0, 1 or 2; and c plus b is not greater than 3.

This group can be part of a monomer aminoalkylalcoxysilane, of an aminoalkylpolysiloxane, or of a copolymer or of a simple mixture of an aminoalkylpolysiloxane with one or more other siloxanes. It is not essential to apply these materials in pure form, and crude hydrolysates or aqueous or alcoholic solutions of the silicon compound can be used.

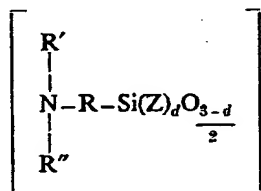
The aminoalkylalcoxysilanes which can be used can be represented generally by the following formula:



in which R, R' and R'' have the same meaning as above; X is an alkoxy radical; Y is an alkyl or aryl radical; c is equal to 1 or 2; b is equal to 0, 1 or 2; and the sum of c and b is not greater than 3 and preferably not greater than 2.

The aminoalkylalcoxysilanes of the type above and the processes for production of compounds with this structure are described in a general manner in French Patent Nos. 1140301 and 1184096. Furthermore, silanes which contain two amino groups can be prepared by reacting a diamine with the appropriate chloralkylalcoxysilane. When the monomer silanes are put in aqueous solution, the alkoxy groups are slowly hydrolyzed in such a way that the monomer silanes are finally converted into aminoalkylpolysiloxanes which are soluble in water. Aqueous mixtures of these polysiloxanes with water soluble organic compounds have good stability.

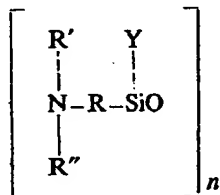
The aminoalkylpolysiloxanes which can be used for implementation of the invention can be straight, cyclic or crosslinked. The crosslinked aminoalkylpolysiloxanes are easily obtained by hydrolysis and then condensation of trialcoxy-substituted silanes. It is possible to represent the polysiloxanes of the above types in a general manner by the following structural formula:



in which R, R' and R'' have the same meaning as above; Z represents hydroxyl or alkoxy groups; and (d) has an average value between 0 and 2, and preferably between 0 and 1. As a typical example of these polymers, it is possible to mention gamma-aminopropylpolysiloxane and

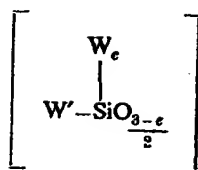
delta-amino-butylpolysiloxane and hydrolysates containing hydroxyl and alkoxy groups and condensates of these polymers.

Aminoalkylpolysiloxanes of the cyclic and straight types are obtained by hydrolysis and condensation of dialkoxy-substituted silanes. It is possible to represent these polymers in a general manner by the following structural formula:



in which R, R' and R'' have the same meaning as above; Y is an alkyl or aryl radical; and (n) is a whole number with a value of at least 3, with average values between 3 and 7 for the cyclic polysiloxanes and higher values for the straight polysiloxanes. As typical examples of cyclic polymers, it is possible to mention the cyclic tetramers of gamma-aminopropylmethylpolysiloxane and of delta-aminobutylmethylpolysiloxane. The straight polymers include gamma-aminopropylmethylpolysiloxane, gamma-aminopropylethylpolysiloxane, delta-aminobutylmethylpolysiloxane, gamma-aminobutylmethylpolysiloxane, and polysiloxanes blocked at the ends in which 1 to 3 alkyl, aryl, alkoxy or hydroxy groups are bound to the terminal silicon atoms of the molecules forming the polymer chain.

The copolymer polysiloxanes which can be used according to the invention can contain siloxane units consisting of one or another of the typical groups described above, in combination with one or more other siloxane units substituted by a hydrocarbon group which is represented by the formula:



in which W and W' are hydrocarbon radicals; and e is a whole number with a value between 0 and 2. It is possible to obtain these copolymers by co-hydrolysis and condensation of aminoalkyl-silanes with other silanes substituted by a hydrocarbon group, or by direct balancing of a mixture of aminoalkylpolysiloxanes and other siloxanes substituted by a hydrocarbon group.

It is also possible to use the aminoalkyl-silicon compounds in the form of their coordinated complexes with metals such as copper, chromium and cobalt. For example, it is possible to prepare copper complexes by reacting the silicon compounds with compounds such as copper chloride, acetate, sulfate, hydroxide or stearate. These complexes can be prepared beforehand or in situ in the treatment bath.

Particular examples of aminoalkyl-silicon compounds are indicated in Table I below; these compounds are identified by a reference number which is used again in the examples.

Table I

Dye fixing agent, No.	Compound or composition
1	Gamma-aminopropyltriethoxysilane;
2	Delta-aminobutyltriethoxysilane;
3	Copolymer of gamma-aminopropyltriethoxysilane and phenyltriethoxysilane (30% solids);
4	30% solution in ethanol of homopolymer gamma-aminopropylpolysiloxane (30% solids);
5	Gamma-aminopropylmethyldiethoxysilane;
6	Copolymer of gamma-aminopropyltriethoxysilane and phenyltriethoxysilane (30% solution in ethanol)
7	N-phenyl-N-methyl-gamma-aminopropyltriethoxysilane;
8	Copolymer silicone oil containing 95.2% dimethylsiloxane blocked at the ends by a trimethylsiloxy group and 4.8% delta-aminobutylmethylsiloxy group;
9	Homopolymer of delta-aminobutylmethylpolysiloxane;
10	Copolymer silicone oil containing 75% dimethylsiloxane blocked at the ends by a trimethylsiloxy group and 25% delta-aminobutylmethylsiloxy groups;

- 11 Copolymer silicone oil containing gamma-aminopropyltriethoxysilane and vinyltriethoxysilane (25% resinous solids);
- 12 Copolymer silicone oil containing gamma-aminopropyltriethoxysilane and amyltriethoxysilane (30% resinous solids);
- 13 Chelate of gamma-aminopropyltriethoxysilane cobalt (17% in H₂O);
- 14 Copolymer silicone oil containing 83.3% dimethylsiloxane blocked at the ends by a trimethylsiloxy group and 16.7% aminopropylsiloxy groups;
- 15 Gamma-aminopropylpolysiloxane; homopolymer coming from gamma-aminopropyltriethoxysilane (50% solids in ethanol);
- 16 N-naphtyl-gamma-aminopropyltriethoxysilane;
- 17 Copolymer containing 50% dimethylsiloxane blocked at the ends by a trimethylsiloxy group and 50% delta-aminobutylmethylsiloxy groups;
- 18 Copolymer containing 70% dimethylsiloxane blocked at the ends by a trimethylsiloxy group and 30% N,N-bis-(beta-hydroxyethyl)-delta-aminobutylmethylsiloxy groups;
- 19 Copolymer containing 27% dimethylsiloxane blocked at the ends by a trimethylsiloxy group, 40% diphenylsiloxy groups and 33% delta-aminobutylmethylsiloxy groups;
- 20 Copolymer containing 68.5% dimethylsiloxane blocked at the ends by a trimethylsiloxy group, 25% diphenylsiloxy groups and 6.5% delta-aminobutylmethylsiloxy groups;
- 21 N-gamma-triethoxysilylpropylpyrrolidene hydrochloride;
- 22 N-beta-cyanoethyl-delta-aminobutyltriethoxysilane
- 23 N,N-dimethyl-gamma-aminopropyltriethoxysilane hydroiodide;
- 24 Beta-methyl-gamma-aminopropyltriethoxysilane;
- 25 Bis(beta-methyltriethoxysilylpropyl)imine;
- 26 N-methyl-beta-methyl-gamma-aminopropyltriethoxysilane;
- 27 N-beta-carbethoxyethyl-gamma-aminopropyltriethoxysilane;
- 28 N-beta-cyanoethyl-delta-aminobutylolysiloxane (mainly cyclic);
- 29 N-beta-furfuryl-gamma-aminopropyltriethoxysilane;
- 30 Delta-aminobutylmethyldiethoxysilane;
- 31 Delta-aminobutylmethylpolysiloxane (crude product otherwise comparable to 9 above - manufactured by hydrolysis, without solvent, of 30);
- [32] Analogous to 31 except that it is manufactured by hydrolysis with the aid of a solvent;
- 33 Delta-aminobutylmethylpolysiloxane incompletely condensed and thus probably containing ethoxy or hydroxyl groups bound to the silicon atom (60% solids in ethanol);
- 34 Aminomethyltriethoxysilane;
- 35 N-beta-aminoethyl-gamma-aminopropyltriethoxysilane;
- 36 Copolymer containing 60% dimethylsiloxane blocked at the ends by a trimethylsiloxy group and 40% N-beta-aminoethyl-gamma-aminoisobutylmethylsiloxy group;
- 37 N,N-bis-beta-hydroxypopyl-gamma-aminopropylpolysiloxane;

- | | |
|----|--|
| 38 | N,N-bis-beta-hydroxystearyl-gamma-aminoisobutylmethyldiethoxysilane; |
| 39 | N-octyl-gamma-aminoisobutylmethyldiethoxysilane. |

Example 1

One immerses samples of cotton cloth boiled and bleached in an autoclave in a dye bath containing 1 wt% "Metromine RF Rubine 3 BLL" (Pr. 491), a direct dye, at 82°C. The ratio of the volume of the bath to the volume of fabric is 40:1. After 20 min, 15% sodium chloride based on the weight of the fabric is added in order to increase the usage of the dye from the bath and application of it to the fabric. After another 15 min, the dyed samples are removed and rinsed with water.

The dyed cotton samples are placed in a series of solutions of equal parts water and isopropanol containing 1 wt% dye fixing agent Nos. 1, 4, 8, 9 and 10 (Table I). Each of these solutions also contains 1% acetic acid. The solutions and the samples are stirred for 15 min at 20°C, and the samples are then removed, rinsed with cold water and dried for 5 min at 121°C. After drying, a piece of white cotton was attached to each of the treated samples. These samples were then individually immersed in water at 71°C, stirred for 1 h, and then removed, rinsed and dried. The quality of color deposited on the white fabric and the color of the immersion water for each of the treated samples was compared with that observed with a dyed sample which was not treated afterwards by a dye fixing agent. In all cases, the aminoalkyl-silicon compounds improved the fastness of the dye. The fastness with regard to washing of the fabric samples runs from the untreated control (the most mediocre) to the fabrics treated by fixing agents 1, 4, 9 and 10 to the fabric treated by fixing agent 8 (the best).

Example 2

One treats five viscose rayon fabrics dyed beforehand in a mixer using a red, turquoise, navy blue, brown and copper brown direct dye in the case of dye fixing agent Nos. 1, 3, 4, 8, 9, 10, 11 and 12. In the course of these treatments, a solution containing 5% of each of the dye fixing agents in a 50:50 mixture of water and isopropanol containing 5% acetic acid is prepared. The fabric samples were padded using each dye fixing solution in order to obtain a moisture absorption of 65%, and they were dried for 5 min at 160°C.

After drying, the fastness with regard to washing of the dyes was determined by the process of Example 1. A sample of each dyed fabric which was not treated by a dye fixing agent was also tested. The comparative results are indicated in descending order; the most mediocre fastness with regard to washing of each series is found at the top of the column and the best is found at the base.

①	②	③	④	⑤
Colorant turquoise non traité	Colorant brun non traité	Colorant brun cuivre non traité	Colorant rouge non traité	Colorant bleu marine non traité
11	4	11	1	8
9	3	4	3	11
3	12	3	11	4
4	11	9	9	3
12	9	12	12	1
1	8	1	4	9
10	10	10	10	12
8	1	8	8	10

Key: 1 Turquoise dye not treated
 2 Brown dye not treated
 3 Copper brown dye not treated
 4 Red dye not treated
 5 Navy blue dye not treated

Example 3

Dye fixing agent No. 13 was applied to rayon substrates direct dyed brown and navy blue with a concentration of 1% padding solids and at 60% moisture absorption; then, they were dried for 10 min at 149°C. On the brown fabric, this dye fixing agent gives excellent dye fixing, while on the fabric dyed navy blue, the dye fixing properties are slightly less effective.

Example 4

In order to compare the dye fixing properties of dye fixing agent No. 9 with that of a dye fixing agent based on commercial amine resin, the following direct dyes were used to color viscose rayon samples:

Yellow "superlite solid yellow EFC" (Pr. 629)

Orange "superlite solid orange LLLWF" (Pr. 628)

Red "superlite solid red 3BL" (Pr. 491)

Blue "superlite solid blue GL" (Pr. 611)

The dye baths contain 2% dye based on the weight of the fabric samples. The ratio of the volume of the bath to the volume of the fabric is 30:1. Each dye is put in paste form using small quantities of sodium alkyl-sulfate as a wetting agent before addition of water. The wetted fabric samples are put in the dye baths and then the temperature is gradually brought to 82°C. After

15 min, 7.5 % Glauber salt ($\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$) is added based on the weight of the fabric to each dye bath. The dyeing is continued for another 15 min, an additional 7.5% Glauber salt is added, and then the dyeing is continued for another 15 min. The samples are then removed from the bath, rinsed with cold water and dried.

The following solutions are applied to each of the dyed fabrics above for the purpose of dye fixing tests:

- A) Control-none;
- B) 0.5% of dye fixing agent No. 9 plus 1% acetic acid in water;
- C) 0.5% of commercial dye fixing agent in water.

The samples are padded with 60% moisture absorption in order to deposit 0.3% of the dye fixing agent on the basis of the weight of the fabric. They are then dried and cooked for 10 min at 149°C.

The loss of dye was evaluated as was the formation of spots on the cotton fabric attached to the sample as described in Example 1 on the following basis:

- Assessment of 4 = extensive loss of dye and formation of spots;
- " of 3 = average loss of dye and formation of spots
 - " of 2 = slight loss of dye and formation of spots;
 - " of 1 = very slight loss of dye and formation of spots;
 - " of 0 = no loss of dye or formation of spots.

The assessments of the samples are listed in Table II below and reveal that dye fixing agent No. 9 is superior to the commercial dye fixing agent under the conditions of this test.

Table II. Assessment of loss of dye and formation of spots

① Colorant appliqué (Abrégé)		② Témoin non traité		⑤ Agent fixateur de colorant, N° 9		⑥ du commerce	
		③ Perte	④ Taches	③ Perte	④ Taches	③ Perte	④ Taches
⑦ Jaune		4	4	0	0	1	1
⑧ Orange		4	4	0	0	3	3
⑨ Rouge		4	4	0	0	1	0
⑩ Bleu		4	4	1	0	3	3

Key:	1	Dye applied (abbreviated)
	2	Untreated control
	3	Loss
	4	Spots
	5	Dye fixing agent, No. 9
	6	Commercial
	7	Yellow
	8	Orange
	9	Red
	10	Blue

Example 5

A—A group of different dye fixing agents was compared using samples of a rayon fabric direct dyed brown using 1% solids in a padding bath with a moisture absorption of 60% in order to deposit 0.6% solids, after which the usual drying and the tests of loss of dye in hot water are done. The results are indicated in Table III hereafter based on the assessment scale of Example 4. Dye fixing agent Nos. 18, 19 and 23 were the most effective. (It should be noted that these three fixing agents contain primary, secondary and tertiary amino groups.) The silanes based on aromatic phenyl- and naphthyl-amine (dye fixing agent Nos. 7 and 16 respectively) were the least effective at this concentration.

Table III

① Agent fixateur de colorant	② Perte de colorant et formation de taches
Non traité ③	4
N° 7	4
N° 16	3
N° 20	2
N° 22	2
N° 18	1
du commerce ④	0
N° 23	0
N° 19	0

Key: 1 Dye fixing agent
 2 Loss of dye and formation of spots
 3 Untreated
 4 Commercial

B—Additional dye fixing tests were conducted using 0.5% of dye fixing agent Nos. 9, 10 and 17 in the padding bath also containing 1% acetic acid. A fabric dyed using the four different direct dyes was treated by each fixing agent.

The results are listed in Table IV below based on the assessment scale of Example 4. The results show that dye fixing agent No. 10 is overall only slightly superior to the commercial fixing agent (an amine resin), while both Nos. 9 and 17 are superior to the commercial fixing agent. Moreover, although none of the dye fixing agents based on aminoalkyl-silicone [sic; silicon] causes a change of shade of the scarlet fabric, the commercial dye fixing agent gives a blue maroon color.

Table IV

Agent fixateur de colorant utilisé (1)	Colorant (2)	Perte de colorant et formation de taches (3)
(4) Néant	Turquoise	4
(5) du commerce	—	2
N° 10	—	0
N° 17	—	0
N° 9	—	0
(4) Néant	(6) Brun	4
(5) du commerce	—	2
N° 10	—	3
N° 17	—	1
N° 9	—	0
(4) Néant	(7) Cuivre	4
(5) du commerce	—	3
N° 10	—	2
N° 17	—	1
N° 9	—	0
(4) Néant	(8) Ecarlate	4
(5) du commerce	—	3
N° 10	—	2
N° 17	—	0
N° 9	—	0

Key:	1	Dye fixing agent which was used
	2	Dye
	3	Loss of dye and formation of spots
	4	None
	5	Commercial
	6	Brown
	7	Copper
	8	Scarlet

C—Other dye fixing tests were conducted with dye fixing agent Nos. 5, 9, 14 and 15, in the course of which 1% of each product was applied from solutions in a 50:50 mixture of water and isopropanol containing 1% acetic acid. The results are indicated in Table V below based on the assessment scale of Example 4.

These results reveal that a polymer of a silane (No. 15) is much more effective than the monomers from which it was prepared (No. 5).

Table V

Agent fixateur de colorant utilisé (1)	Colorant (2)	Perte de colorant et formation de taches (3)
Néant (4)	Ecarlate (5)	4
N° 5	—	4
N° 14	—	3
N° 15	—	0
N° 9	—	0
Néant (4)	Bleu marine (6)	4
N° 5	—	3
N° 14	—	3
N° 15	—	2
N° 9	—	0
Néant (4)	Brun (7)	4
N° 5	—	3
N° 14	—	2
N° 15	—	1
N° 9	—	0
Néant (4)	Turquoise	4
N° 5	—	3
N° 14	—	1
N° 15	—	1
N° 9	—	0

Key:	1	Dye fixing agent which was used
	2	Dye
	3	Loss of dye and formation of spots
	4	None
	5	Scarlet
	6	Navy blue
	7	Brown

Example 6

A series of crude hydrolysates of aminoalkyl-silicon compounds was tested as to the dye effectiveness. In the course of these tests, the fixing agents were prepared in the form of aqueous solutions. All the aqueous solutions were left at rest for 30 min before applying them to the fabric.

Group I:

- 1) 0.5% dye fixing agent No. 9 + 1% acetic acid;
- 2) 0.5% dye fixing agent No. 31 + 1% acetic acid;

- 3) 0.5% dye fixing agent No. 32 + 1% acetic acid;
- 4) 0.5% dye fixing agent No. 33 + 1% acetic acid;
- 5) 0.5% dye fixing agent No. 30 + 1% acetic acid;
- 6) 0.5% dye fixing agent No. 2 + 1% acetic acid;
- 7) 0.5% a dye fixing agent based on commercial amine resin.

These solutions were applied to rayon fabrics direct dyed by padding with a moisture absorption of 60%, and they were then dried for 10 min at 149°C.

Fabrics were tested as to the loss of dye and fastness with regard to washing by immersing them for one hour at 71°C in water containing 0.1% sodium alkyl sulfate as a wetting agent. The baths were periodically stirred. A piece of white cotton fabric was attached to each dyed sample in order to determine the transfer of dye. The samples were dried in the air at 20°C. The loss of color and the transfer of color to the white fabric were assessed based on the assessment scale defined in Example 4.

The results are indicated in Table VI below. It was observed that the three dye fixing agents of the crude hydrolysate type (Nos. 31, 32 and 33) are identical as to their yield with the pure aminoalkyl-silicon compound No. 9 and are as good as if not better than the dye fixing agent based on commercial amine resin. The monomer dye fixing agents (Nos. 21 and 30) were slightly inferior to the polymers and display a variable behavior over the four different direct dyed fabrics.

Table VI

① Agent fixateur de colorant utilisé (Echantillons 1 à 7)	② Degré de perte de colorant et formation de taches			
	Turquoise	Rouge ③	Cuivre ④	Brun ⑤
⑥ Néant	4	4	4	4
N° 9	0	0	0	0
N° 31	0	0	0	0
N° 32	0	0	0	0
N° 33	1	0	0	0
N° 30	1	4	2	2
N° 2	0	4	3	3
⑦ du commerce	1	0	1	0

Key: 1 Dye fixing agent which was used (Samples 1 to 7)
2 Degree of loss of dye and formation of spots

- 3 Red
- 4 Copper
- 5 Brown
- 6 None
- 7 Commercial

A similar series of tests in which cupric chloride was used to complex the dye fixing agents based on silicon classified the fixing agents in the same order.

Example 7

Dye fixing agent Nos. 24, 25, 26, 27, 28, 29, 30 and 34 (Table I) were tested as to their dye fixing effectiveness on four rayon fabrics direct dyed using 1% fixing agent and 1% acetic acid in the solutions. In each case, the solutions were padded on dyed fabrics which were then dried for 10 min at 149°C. The fabric was then immersed in water at 71°C for 1 h in order to assess the dye fixing effectiveness. The assessments, using the scale of Example 4, are indicated in Table VII. Changes of shade in the test made assessment of the samples difficult, but it is obvious that all the aminoalkyl-silicon compounds were effective at least to a certain degree. The effectiveness extends from that of No. 28 to that of the aminoethyl compound (No. 34).

Table VII

Agent fixateur de colorant utilisé (1)	Degré de perte de colorant et formation de taches (2)			
	Turquoise	Rouge (3)	Cuivre (4)	Brun (5)
(6) Néant	4	4	4	4
N° 34	3	3	3	3
N° 24	2	2	3	2
N° 25	0	2	3	2
N° 26	2	2	3	2
N° 27	1	2	2	2
N° 28	0	0	0	0
N° 29	0	2	3	2
N° 30	1	4	3	2

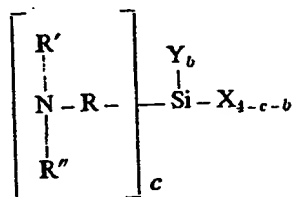
- Key:
- 1 Dye fixing agent which was used
 - 2 Degree of loss of dye and formation of spots
 - 3 Red
 - 4 Copper
 - 5 Brown
 - 6 None

Claim

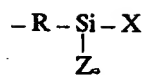
A process for improving the fastness of dyes or prints on textile articles, characterized by the fact that one applies to said articles, [which are] dyed or printed beforehand, an aminoalkyl-silicon compound or a coordinated metallic complex of the latter.

Subordinate claims

1. A process according to the claim, characterized by the fact that the complex of the silicon compound is coordinated with copper or chromium.
2. A process according to the claim, characterized by the fact that the article is made of cellulose fibers.
3. A process according to the claim, characterized by the fact that the aminoalkyl-silicon or its complex is applied to the textile article using a solution or suspension in water, in an alcohol or in a mixture of water and alcohol.
4. A process according to the claim, characterized by the fact that the textile article is heated to a high temperature after having applied the aminoalkyl-silicon compound or its complex.
5. A process according to subordinate Claim 3, characterized by the fact that the solution or suspension of the aminoalkyl-silicon compound contains an acid.
6. A process according to subordinate Claim 5, characterized by the fact that the acid is a monobasic organic acid.
7. A process according to the claim, characterized by the fact that the aminoalkyl-silicon compound contains at least one functional group with formula:



in which R is a bivalent hydrocarbon residue with at least three carbon atoms between the nitrogen atom and the silicon atom, R' and R'' represent hydrogen atoms or alkyl, aminoalkyl, cyanoalkyl, hydroxyalkyl, carboxyalkyl, carboalcoxyalkyl, or aryl radicals, or the monovalent group:



X is an alkoxy radical or the oxygen atom of a siloxylidene radical $[-O-Si\equiv]$; Y is a hydroxy, alkyl or aryl radical; Z is an alkoxy, alkyl or aryl radical; c is equal to 1 or 2, b is equal to 0, 1 or 2; and c plus b is not greater than 3.

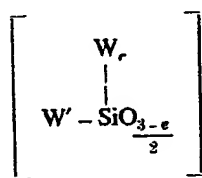
8. A process according to subordinate Claim 7, characterized by the fact that X is an alkoxy radical and Y is an alkyl or aryl radical.

9. A process according to subordinate Claim 7, characterized by the fact that the aminoalkyl-silicon compound is a polysiloxane containing units corresponding to the above-mentioned formula in which X is an oxygen atom of a siloxylidene radical $(-O-Si\equiv)$.

10. A process according to subordinate Claim 9, characterized by the fact that Y is an alkoxy or hydroxy radical.

11. A process according to subordinate Claim 9, characterized by the fact that Y is an alkyl or aryl radical, c is equal to 2 and at least three of said units are joined by silicon-oxygen-silicon bonds.

12. A process according to subordinate Claim 9, characterized by the fact that the polysiloxane also contains at least one unit with formula:



in which W and W' are hydrocarbon radicals; and e is a whole number with the value between 0 and 2.

13. A process according to the claim, characterized by the fact that the aminoalkyl-silicon compound is delta-aminobutylmethylsiloxane, a copolymer siloxane containing a dimethylsiloxane blocked at the ends by a trimethylsiloxy group and delta-aminobutylmethylsiloxy groups or N-beta-aminoethyl-gamma-aminoisobutylmethylsiloxy

groups, N-beta-cyanoethyl-delta-aminobutylmethylpolysiloxane, or a crude hydrolysate of delta-aminobutylmethylpolysiloxane.

14. A process according to the claim, characterized by the fact that the aminoalkyl-silicon compound is applied to the textile article using a solution containing approximately equal parts of water and isopropanol and a small quantity of acetic acid.

15. A process according to subordinate Claim 7, characterized by the fact that the aminoalkyl-silicon compound is N-beta-aminoethyl-gamma-aminopropyltrimethoxysilane.